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MATERIALS RESEARCH AT UNIVERSITIES--EN FRANCE ET/UND IN DEUTSCHLAND

ABRAHAM SOSIN

31 MARCH 1977

UNITED STATES OF AMERICA

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER 14 ONRL-R-2-77	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) 6 MATERIALS RESEARCH AT UNIVERSITIES--EN FRANCE ET/UND IN DEUTSCHLAND		5. TYPE OF REPORT & PERIOD COVERED
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) 10 Abraham Dr. A. SOSIN		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS OFFICE OF NAVAL RESEARCH, BRANCH OFFICE LONDON BOX 39, FPO NEW YORK 09510		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS		11 12. REPORT DATE 31 Mar[redacted]77
		13. NUMBER OF PAGES 11 12 14p.
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) UNCLASSIFIED
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

APPROVED FOR PUBLIC RELEASE: DISTRIBUTION UNLIMITED

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

265 000

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

			Irradiation
Mossbauer Spectroscopy	Myogloben	Ion-implantation	Amorphous
Ferroelectrics	Hemoglobin	Dislocations	Annealing
Magneto state	Grain Boundary Energy	Fatigue	Vacancies
Iron	Polyethylene	Spin-glass	Diamond
Textures (metallurgical)	Mechanical Properties	Silicon	Interstitials

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Research activities in materials science and in solid state physics at two German and two French universities have been reviewed. These universities are the University of Saarlandes, the University of Karlsruhe, the University Louis Pasteur (Strasbourg), and the University of Paris. An account of some of these research activities is given. In addition, we present a discussion of the status of research activities and the stresses imposed on these activities.

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S/N 0102-014-6601

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MATERIALS RESEARCH AT UNIVERSITIES--EN FRANCE ET/UND IN DEUTSCHLAND

I. Introduction.

Attempting to assess the status of research into materials science in a nation's universities may be a dangerous enterprise, for time does not allow an exhaustive study and sampling may be unrepresentative. In hopes of making observations more representative, I decided to visit universities in France and West Germany that are not, I thought, considered among the front-runners in materials research in their countries, and then I added one that is. As matters developed, my selection did not match my intent but seemed to provide much of the survey I sought.

II. University of Saarlandes (Saarbrücken, W. Germany).

The scope of the research work in materials here was well beyond my expectations. By their (Saarlandes' professors) own estimate--which I judge to be accurate--materials research at this University ranks among the first four in Germany. According to this ranking, the other top institutions would be the Universities of Göttingen, Erlangen and Stuttgart.

The University of Saarlandes is of recent vintage, constructed on a former military installation some 20 years ago. The architecture is a mix of new buildings plus some remaining (rather sterile) military buildings. The facilities for research are excellent. While a concern does exist for some possible retrenchment in financial support, the mood is progressive. Of more importance than direct financial endowment are two other phenomena. First, the "Institut System" still prevails, albeit slightly diminished. In times of expansion, able young researchers were accommodated by new Institutes. But this is largely history, so Germany is troubled about the possibility of losing a major portion of the newer generation of scientists. (A partial solution to this problem in France is described below.) Second, career protection means that staff personnel (particularly non-technical staff, for the discussion here) are hired seemingly forever (as is true in France, too). Any retrenchments or accommodations for inflation (which is relatively small in Germany) are achieved by cuts in student and project funds. State funding for basic research capabilities remains adequate so far.

While these matters hang over Saarlandes (and all W. German universities) as a small black cloud, generally the work basks in bright sunlight. Materials research is carried out in several departments drawn together in a loose confederacy. In addition, there is a field-of-expertise umbrella grant in ferroelectrics which spans several professorial institutes. This grant, at the 1-2 x 10^6 DM per year level, was recently extended for three years by the University.

The area ~~of a~~ particular Institute generally covers one or more disciplinary themes. In the case of U. Gonser's Institute, the activities revolve about an experimental tool--Mössbauer resonance spectroscopy (MRS). Indeed, the range and varieties of subjects addressed by Gonser and coworkers in MRS is spectacular. They have contributed to ferroelectric research, using MRS, with studies of the spin-states in Co- and Fe-doped $LiNbO_3$, the latter being a candidate ferroelectric medium for volume holography, and have shown that the Fe^{2+}/Fe^{3+} ratio can be altered by oxidation or reduction heat treatments. Using a polarized x-ray technique, they determined the direction of the principal axis of the electric field gradient of the Fe^{2+} site. This work has also included Fe-doped $LiTaO_3$, $BaFe_{12}O_{19}$, As-substititued $BaFe_{12}O_{19}$, etc. Non-oxides investigated include FeB and FeV. These

studies might be termed as truly microscopic, working at local environments near Fe ions.

In MRS, ^{57}Fe serves as the dominant probe, so it is logical that Gonser has directed attention to Fe-containing material. One such material is Fe itself, and a question that Gonser *et al* have examined is the magnetic state γ-Fe. γ-Fe is unstable with respect to α-Fe at lower temperatures, but coherent γ-Fe particles can be stabilized with less than 1% Cu (which is diamagnetic). Gonser *et al* used Cu-Fe alloys with precipitated γ-Fe particle sizes between 25 Å and 350 Å and measured the magnetic transition (Neel) temperature. For particle sizes ∿300 Å, T_N = 57 K; below 300 Å, T_N drops to zero as the size tends toward zero (as it must). The use of MRS allows a measurement of the average internal magnetic field, H_o, in γ-Fe, 23 kOe; for comparison, H_o = 330 kOe in γ-Fe.

Other studies of MRS in metallic systems include: (A) MRS in Al-0.5-5/%Fe, splat-quenched. The spectra show enhanced quadrupole splitting due to Fe associations (e.g., Fe dimers). (B) MRS in Fe-28%Ni-3%C--invar. The spins of the large magnetic field components align colinear to an applied external-field direction, but the hyperfine field of the lower-field components align perpendicular, indicating the coexistence of some anti-ferromagnetic component along with the standard ferromagnetic one. (C) The use of MRS in texture determinations. Texture is a standard metallurgical problem, and the use of MRS here is of great potential value. Unfortunately, anisotropy of lattice vibrations (the Goldanskii-Karyagin effect) also results in MRS hyperfine pattern variations which confuse texture determinations. (D) MRS in myoglobin, deoxymyoglobin, and bacterial cotalase. These studies, similar to previous ones by Gonser on hemoglobin, indicate that internal structure of biological molecules can be elucidated by MRS. (E) MRS study of magnetic recording tapes (i.e., thin Fe_2O_3 films on polymer substrates). Here Gonser *et al* were able to determine the amount (16%) of super-paramagnetic component present, a component which is useless for practical purposes, and the particle alignment available. They found an alignment parameter of 1.1 compared with an ideal of 1.33, demonstrating considerable alignment but far from ideal.

Imaginative "metallurgy" is the research area of H. Gleiter and colleagues. In more conventional metallurgy, they have considered an old problem--grain boundary energy determinations, as a function of mismatch, angle between grains in metals--using a novel method to avoid the many samples and multitude of measurements. Gleiter does, in fact, use many samples and a multitude of measurements, but in an ingenious way which drastically reduces the drudge. He starts with a crystallographically-oriented (001) single crystal sheet, for example, then places on this sheet 10^4 single crystal spheres, each

of about 100 m diameter. Their orientations are determined, *en masse*, by x-ray diffraction. Obviously the spheres are positioned at random initially, which generally means high-energy contacts. The spheres rotate toward lower-energy orientation when the ensemble is heated, typically for about 100 hours up to temperatures within 1 degree of melting; they also rotate upon the application of pressures (up to 30 kbar). Among the observations that Gleiter *et al* have made is that the grain boundary energy, in Cu-Ni, depends on the electron-to-atom ratio.

Polyethylene is "metal" that this group has examined in some detail. In a study of thin molten polyethylene films, J. Petermann and Gleiter have used defocus-contrast transmission electron microscopy. They observe a pattern of quasi-parallel crystals, each about 400-500 Å thick, separated by thin amorphous regions before melting, the crystals aligned approximately parallel to the direction of the drawing that was used in producing the films. On melting (about 150°C) the initially sharp amorphous region-crystal contrast is reduced, but considerable structure remains. If the film is electron-irradiated (in the microscope), the resulting pattern is very similar to the original one. Petermann and Gleiter argue that this lends weight to the identification of the molten film as a smectic liquid crystal, where each layer is formed by folded molecules whose chain axes lie approximately parallel to the direction of drawing. Upon raising the temperature from room temperature to 130°C, the amorphous region increased over two-fold. Petermann and Gleiter have also looked at recrystallization in polyethylene at free surfaces and find the process to be basically the same as occurs in metals, suggesting that metallurgical concepts can be profitably carried over to polymers---at least to semi-crystalline ones.

III. The Institut für Werkstoffekunde at the Universität Karlsruhe.

This Institute, under the direction of E. Macherauch, is cast in the more conventional mold of metallurgy departments. Some efforts to extend the scope to include polymers have been largely unsuccessful; metals are the central theme, and attention is restricted largely to steels and copper alloys. A possible advantage of this counter-current posture, in a time when materials science appears to be *à la mode*, is the financial support that the Institute receives from organizations that identify closely with metals and associated processes: welding, casting, etc.

Measurements of mechanical properties (e.g., stress-strain curves) dominate research activities. For example, dynamic tensile tests of α-Cu (Al, Ga, or Ge) alloys have been run at various strain rates and in stress-relaxation tests in the temperature range between 78 K and 363 K. The data are analyzed to yield the free activation enthalpy for the interaction of glide dislocations with solute atoms. The enthalpies vary from 1.2 to 1.9 eV, depending on solute type and

concentration (1.22 eV for 3.4% Ga vs 1.67 for 1.2% Ga). The quantity monitored in this work was the stress for 0.2 strain offset. The serrated yielding phenomenon, the Portevin-Le Chatelier Effect, has also been examined as a thermally activated process. This effect is linked with interstitial diffusion in steels, but in copper-based alloys, the Karlsruhe investigators identify the elastic interaction between glide dislocations and solute atoms enhanced by non-equilibrium vacancies produced by plastic deformation or simply in thermal equilibrium. Under these assumptions, the Portevin-Le Chatelier studies allow the deduction of vacancy-solute atom binding energies, and Macherauch *et al* have studied Cu with additions of Zn, ZnNi, Sn, Al, Ga, Ge, As, and In.

Related studies include the influence of grain size and texture in α-Cu(Zn) and the twinning in α-Cu (Zn, Ga, As, Sn). A critical stress was identified for twinning; this stress is approximately proportioned to the square root of the stacking-fault energy (adjusted by alloying) and the inverse square root of the grain size. The latter relation follows a prediction of the much-used Petch grain-size equation.

The studies in steel, under H. Wohlfahrt, concern processing in technological situations. He has studied fatigue of an annealed low-carbon steel as influenced by the sample preparation method--up-cut and down-cut milling. Since surface conditions are thought to dominate fatigue fracture, Wohlfahrt determined the residual stress state at and near (<60 μm) the surface by x-rays. Although the same hardness was found in either milled treatment, the stress pattern differed entirely; a tensile stress of +210 N/mm^2 was measured at the surface of the down-cut surface while -300 N/mm^2 existed at the up-cut surface. Nevertheless, no substantial difference was found in the fatigue tests. This appears to violate the familiar dogma that compressive stresses increase fatigue strength.

IV. The Université Louis Pasteur in Strasbourg.

Materials research here bears a similarity to that at the Universität Saarlandes in that it is divided among several units. There the similarity ends. In Strasbourg, each of the ten units has proceeded quite independently of the others. On 4 November 1976, these autonomous units met as a "Scientific Interest Group--Materials" to consider ways to bring their diverse interests and capabilities into a more integrated whole. The units are as follows: Laboratoire de Minéralogie et Petrographie; Département Sciences des Matériaux de L'ENSCS; Equipe d'Etude des Surfaces par Diffraction Spectrometrie d'Emission; Laboratoire de Catalyse et Chimie des Surfaces; Laboratoire de Structure Electronique des Solides; Laboratoire de Physique des Milieux Condenses; Groupe de Physique Fondamentale et Appliquée; Laboratoire de Spectroscopie et d'Optique du Corps Solide; Laboratoire de Physique des Rayonnements: Semiconducteurs et Cristeux Moleculaires; Centre de Recherche sur les Macromolecules: Groupe de Spectroscopie Moleculaire et Service de l'Etat Solide; and Laboratoire d'Acoustique Moleculaire.

It is clear that a substantial amount of expertise exists, particularly when it is realized that each unit comprises an appreciable number of professors, faculty, CNRS (Centre National de la Recherche Scientifique) Fellows, and students. However, the extent of involvement in materials research is not uniform, and there has been little materials engineering, either. Adding to these problems in the path toward consolidation is the current French austerity in the support of research. The result of the meeting, not surprisingly, was limited to a resolution to continue efforts toward consolidation, but few substantive actions.

My observations in Strasbourg were limited to the electronic structure of solids, and I was hosted by E. Daniel. In the tradition of Pierre Weiss, magnetic properties are a recurring theme, but other properties--electrical resistivity, Seebeck coefficient, specific heat, etc.--are included. The theory group is exceptionally strong, and band calculations are emphasized. Transition metals and compounds are receiving the main current attention. The presence of anti-ferromagnetism in TS_2 and TSe_2 (where T = Ni, Co, Fe) and in related ternaries (e.g., TS_xSe_{2-x}) has been examined. "Spin-glasses" are another prime topic. Spin-glasses are metallic alloys in which a magnetic element is dissolved in a non-magnetic matrix (e.g., Fe in Au, Mn in Ag). It is believed that polarization of the conduction electrons occurs around localized moments, via an s-d interaction. This polarization shows spatial oscillations which can lead to ferromagnetic or anti-ferromanetic spin couplings, depending on the separation distance of the spins. In the spin-glasses [at OK and small solute (i.e., spin) concentrations], a freezing of moments occurs, but in a random manner. Only the randomness leads to the inclusion of the term, glass, in the title of the phenomenon. Recent work at Strasbourg on Fe in Mn demonstrates this to be a spin-glass system also, even though the host atom is transitional. Specifically, for Fe concentrations below 0.25%, the Fe atoms are magnetic, non-interacting, and lead to a Kondo effect. For concentrations between 0.25% and 3%, the spin-glass structure exists. For higher concentrations, long- and short-range interactions also occur leading progressively to the dominance of (short-range) ferromagnetic couplings, with the formation of large magnetic clusters leading to super-paramagnetism.

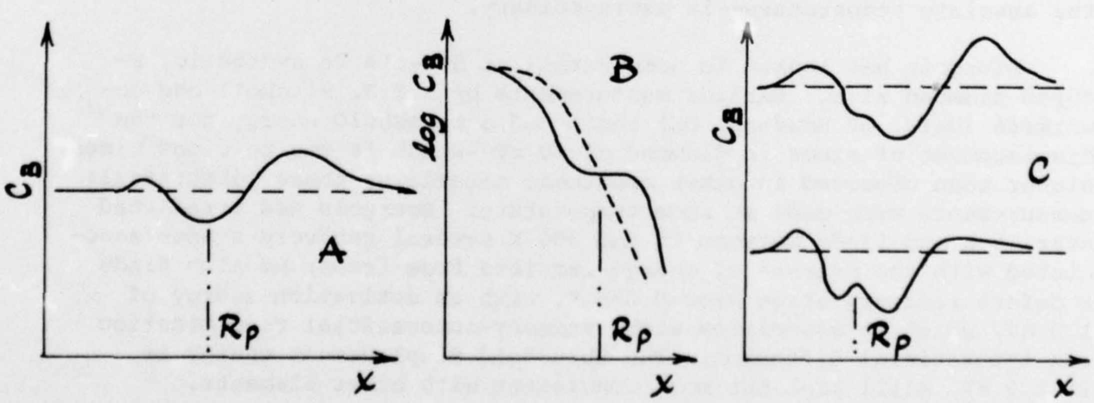
V. The Université de Paris.

This is a multi-campus organization. The majority of solid-state studies takes place on a campus in the lively Latin-Quarter region; on the Orsay campus south of Paris; southwest of Paris in Bellevue; and north of Paris on the new campus of Villetaneuse. The Paris campus was built in the more heraldic days of de Gaulle, and the scope of the architecture is consistent with that spirit: a number of large, cavernous buildings, interconnected below ground and separated by concrete at street level. In space allocation, the campus appears

to have been well-endowed, but aesthetics were largely forgotten, and the situation is not helped by the posters and paintings of a highly politically-conscious student body. Political overtures enter into university affairs, even guiding faculty organization.

If the Paris campus is a "concrete tangle," the Orsay campus is, on the exterior, the reverse, cast in rural beauty and forested isolation. Internally, Orsay buildings are much like the Paris buildings--ample but somewhat dreary. And there are other common denominators for the two campuses--a growing shortage of financial support for research and a substantial lack of students in the sciences.

Beset by these problems, the research accomplishments of the Groupe de Physique des Solides in Paris emerge as impressive. P. Baruch, my host in Paris, continues to elucidate radiation-enhanced diffusion. Most recently he has addressed the problem of the redistribution of B which occurs in Si upon bombardment; he is currently using protons in the 250-450 keV range. Some of the results are as follows: (A) If the b concentration profile, C_B, is initially uniform, irradiation produces a dip and corresponding peaks, both located near the end of the range, R_p, of the protons--where the number of interstitial atoms and lattice vacancies created by the protons is maximal. (B) If the initial B-concentration profile results from a surface prediffusion treatment, the dips and peak are again present and the "up-hill" diffusion is very clear. (C) A clear examination of the case for an initially uniform profile shows a double-dip structure. The profiles are shown schematically below; in Figure C, curve 1 applies for a light proton bombardment, curve 2, for a heavy bombardment.



The double-dip profile is the key to Baruch's model that two mechanisms operate. Si interstitials, created by collisions with high-energy protons, diffuse until meeting B substitutional atoms; on reaction, the Si becomes substitutional and the B, interstitial. B interstitials are fast diffusers and allow B atoms to diffuse extensively until they meet vacancies, also created by bombardment. The reaction of a B interstitial and a Si vacancy yields a B substitutional atom, terminating the process. In the first mechanism, B-transport is wholly as described above; in the second mechanism, B-transport (either interstitially or substitutionally) ends at B clusters which owe their existence to an irradiation-induced local excess of B.

Still other mechanisms of defect diffusion in Si and other semiconductors have been explored by J. Bourgoin, a very active Centre National de la Recherche Scientifique (CNRS) Fellow in Baruch's group, in collaboration with J.W. Corbett (SUNY, Albany). One migration mechanism proposed by Bourgoin starts with the initial presence of a defect in a state of pseudo-equilibrium and, therefore, in an energy trough. Suppose the charge state of the defect is altered by interaction with a bombarding particle or by a photoelectric process. The stability of the defect would be effected so that the potential energy diagram may be inverted, in which case the defect will find itself at an energy peak. This is a temporary configuration. If the charge state is sufficiently persistent, the defect will move--toward a trough in the inverted energy diagram. This model was inspired by an observation made by J. Zizine, also in Baruch's group, in n-type Ge irradiated near 4 K. An annealing stage is observed at 65 K on heating following irradiation when operating in the dark. Under illumination, the stage is shifted to 27 O K. This shift in temperature--over half the absolute temperature--is extraordinary.

Bourgoin has looked in some detail at defects in synthetic, B-doped diamond also. Earlier measurements by E.W.J. Mitchell and co-workers (Univ. of Reading, UK) indicated a threshold energy for the displacement of atoms in diamond of 80 eV--which is two to eight times higher than observed in other monatomic materials; these (electrical) measurements were made at room temperature. Bourgoin has irradiated near 15 K and finds between 15 and 300 K several recovery stages associated with the release of charge-carriers from traps; he also finds a defect recovery stage around 260 K, with an activation energy of 1.3 eV, which he associates with vacancy-interstitial recombination via interstitial diffusion. The threshold displacement energy is 35 ± 5 eV, still high but more consistent with other elements.

Extension of this work has led Bourgoin to the study of B implantation into diamond, as well as the implantations of N and C ions. These studies confirm the applicability of the theory of K.B. Winterbon

for ion ranges. The use of ion-implantation leads to the amorphization of the heavily damaged regions of a material, and Bourgoin has examined the kinetics of crystallization of these amorphous regions in Ge and Si, obtained during ion implantation or by evaporation on a substrate. In Si, amorphized by bombardment of 10^{15} P ions cm^{-2} at 80 keV, Raman spectroscopy was used as the means for study. The (broad) Raman peak near 480 cm^{-1} diminished during post-irradiation isothermal annealing at 500°C, while the (sharp) lattice peak at 522.5 cm^{-1} increased. The time-dependences of the variation of these peak amplitudes provide a history of crystallization which Bourgoin has found to fit a homogeneous first-order rate process, consistent with the passage of a crystalline front through the amorphous region at a rate of 1.6×10^{-2} Å/sec at 550°C. Bourgoin has used electrical conductivity measurements in amorphous Ge with similar success.

Until recently, the radiation effects group of P. Lucasson has been an organizational unit of the Paris campus, located in Orsay to accommodate the physical needs, but a transfer of organization is underway. The work of this group continues to emphasize two phenomena in metals. The anisotropy of defect creation in electron-irradiated metals continues, with an extension to the consideration of recovery of Fe single crystals after irradiation. Examples of the scope of this work in Fe are the following. Minimum energy for displacement of an atom in the <100> direction--17 eV; in the <111> direction-20 eV; in the <100> direction--more than 30 eV. Recovery near 66 K is due to defects created in the <100> direction; recovery near 87 K is probably due to recombination of interstitial-vacancy pairs in the <111> direction. Between 90 K and 110 K, defects produced in the <100> direction dominate at lower temperatures, and those produced in the <111> direction dominate at the higher temperatures.

The second phenomenon is the trapping of interstitial atoms near impurity atoms during interstitial migration. Lucasson and coworkers have examined Al with addition of small concentrations of Mg or Ag, and Ag with small additions of Cd and In. By analyzing the suppression of annihilation of interstitial atoms at vacancies during interstitial diffusion, due to the trapping of the alloying additives, they are able to deduce the capture radius of each solute atom type. The analysis is based on computer fits to the solution of coupled chemical rate-theory equations. Some ambiguity arises (e.g., in AgIn) probably due to the reliance on chemical-rate theory rather than full diffusion analysis. Lucasson is attempting to use many of these techniques in a new area: hydrogen in metals. He has found a shallow minimum in the electrical resistivity vs temperature curves in the case of 23% H in W. The source of the minimum is unknown and, to complicate matters, the extent of the minimum depends on the rate at which the sample is heated in making the measurements through the critical temperature range near 180 K.

VI. Concluding Remarks.

I have already referred to the financial stresses in the French university research system. Part of this is due to actual reductions in fund allocations, coupled with appreciable inflation. Aggravating this is the very tight civil service scheme. Once an employee has been in service for three months, dismissal is almost an impossibility. This frequently forces the principal investigator to support a staff which, in fact, has outgrown its financial base. It is clearly counter-productive in encouraging any expansion; employees who may be hired in a moment of success can linger as an impediment in a leaner (and longer?) era.

The CNRS fellow system enters this picture in an unique way. The fellow is supported by the CNRS; in principle, he has only secondary allegiance to the institution where he works. He may, in point of fact, decide to leave that institution and move elsewhere, with the agreement of the succeeding institution. In doing so, he may take with him the instrumentation procured for him with CNRS funds. Accordingly, the CNRS fellow has traded the security and, perhaps, status of a full university position for the leverage and (currently) superior support for research. There is much to be admired in this approach. There is also a measure of institutional insecurity, as well, and this extends to the individual professors who find themselves beset by still another difficult situation for accommodation.

Thus, one finds that Germany and France share some common elements in their research picture but, overall, show little resemblance. The common elements are national inflation and research support cut-backs. However, the French problems here are so much more severe than in the German case as to make this similarity almost superficial, the German scene being most significantly brighter for research. It takes little subtle detective work to observe the difference. The spirit of the German researchers, from professor to students, is one of involvement and excitement; in France, a spirit of resignation, possibly even despair, comes through (though this is hardly universal).

There is one common element between the French and German research pictures which strikes the American as peculiar if not bordering on the impossible. The case is particularly striking in France. Despite stringencies and problems of other kinds, the equipment in the research laboratories is markedly superior to that encountered in the average American university research laboratory. One could argue that this represents the effects of a short era of glory in French science support during the sixties and that time will take its toll. Perhaps, but new equipment which postdates that wonderful time-span is much in evidence. I rather believe that it is a by-product, certainly in Germany, of the Institute system in which the philosophy is to crown a relatively few individuals, then to support them in a style "befitting

their stations." American science (and engineering) support has emphasized a more common denominator. The merits of the two philosophies--American vs European--and their resulting system provide the basis for an interesting study.